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DEVELOPMENT OF HTPB PROPELLANT FOR BALLISTIC MISSILES

Grant Thompson, et al

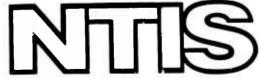
Thiokol Corporation

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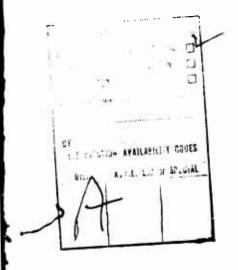
Air Force Rocket Propulsion Laboratory

July 1974

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This is the ninth Quarterly Progress Report prepared by Thiokol Corporation, Wasatch Division, Brigham, City, Utah, on the work accomplished on Contract F04611-72-C-0048 during April through June 1974.

Dr. Grant Thompson is the Principal Investigator and Mr. E. E. Day is the Program Manager, The AFRPL Project Engineer is Mr. Wayne E. Roe (MKPA).

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

For the Commander Charles R. Cooke

SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered) READ INSTRUCTIONS REPORT DOCUMENTATION PAGE BEFORE COMPLETING FORM 1. REPORT NUMBER 2. GOVT ACCESSION NO. 3. RECIPIENT'S CATALOG NUMBER AFRPL-TR-74-44 4. TITLE (and Subtitle) 5. TYPE OF REPORT & PERIOD COVERED QUARTERLY PROGRESS REPORT NO. 9 Interim, Apr-Jun 1974 April-June 1974, Development of HTPB PERFORMING ORG. REPORT NUMBER Propellant for Ballistic Missiles 74493 7. AUTHOR(a) 8. CONTRACT OR GRANT NUMBER(#) Grant Thompson F04611-72-C-0048 Evan E. Dav . PERFORMING ORGANIZATION NAME AND ADDRESS PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Thiokol/Wasatch Division A Division of Thiokol Corporation Brigham City, UT 84302 11. CONTROLLING OFFICE NAME AND ADDRESS 12 REPORT DATE Solid Rocket Division July 1974 Air Force Rocket Propulsion Laboratory 13 NUMBER OF PAGES Edwards AFB, CA 93523
14. MONITORING AGENCY NAME & ADDRESS(II dillerent from Controlling Office) 20 15. SECURITY CLASS. (of this report) NA UNCLASSIFIED 15. DECLASSIFICATION DOWNGRADING SCHEDULE 16 DISTRIBUTION STATEMENT (of this Report) Approved for public release: distribution unlimited 17 DISTRIBUTION STATEMENT (of the abstract entered in Block 20, If different from Report) NA 18. SUPPLEMENTARY NOTES None 19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Instrumented motors Hydroxyl terminated polybutadiene propellant Structural test vehicles Mechanical properties and aging 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report presents Thiokol's ninth quarter progress on Contract F04611-72-C-0048. The program objective is to develop a family of solid propellants based on HTPB (hydroxyl terminated polybutadiene) binder, and demonstrate one optimized propellant formulation by large scale motor testing. Data are presented for Phase II aging. The TU-775/02 structural test vehicle was instrumented and cast with TP-H1139 propellant. The TU-775/03 motor instrumentation is being monitored.

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1.0 INTRODUCTION

This report presents Thiokol's ninth quarter progress on Contract F04611-72-C-0048, "Development of HTPB Propellant for Ballistic Missiles." The program objective is to develop a family of solid propellants for ballistic missiles based on an HTPB (hydroxyl terminated polybutadiene) binder, and to demonstrate one optimized propellant formulation by large scale motor firings.

Specifically, five propellants have been formulated and are being characterized to the point that they will be ready for motor advanced development programs. These five propellants are compatible with two optimized missile systems: (1) a weight constrained small diameter three stage ballistic missile, and (2) a length constrained large diameter ballistic missile. In addition, a formulation which is typical of the entire series was fully defined, characterized, and demonstrated in the static firing of a Third Stage Minuteman III Motor (TU-775/01).

Significant tasks include missile optimization study and formulation tailoring plus definition of all aspects of processing, casting, storing, and handling this family of propellants. Ingredient specifications, processing instructions, and quality control procedures will be prepared. Clear identification of delivered propellint performance capability will be a major program objective. Two additional Third Stage Minuteman III motors have been instrumented and loaded as structural test vehicles (TU-775/02 and TU-775/03).

2.0 PHASE I--PROPELLANT CHARACTERIZATION

Propellant aging is continuing; no aging data have been generated in this period.

3.0 PHASE II--SCALEUP

3.1 UNIAXIAL MECHANICAL PROPERTIES AND AGING

Additional mechanical properties aging data for the 91 percent solids propellant TP-H1141, Mix No. 8627001, are presented in this report. The uniaxial tensile data (Table 1 and Figures 1 to 3) for TP-H1141 propellant aged 4 mo at 135°F indicate that the stress levels have increased overall. Compared to the figures for this propellant at zero time, * strain capability at the higher test temperatures and lower crosshead rates (lower portion of the failure envelope in Figure 1) has diminished by about 9 to 12 strain percent for a given stress level. On the other hand, strain capability at the lower test temperatures and higher crosshead rates (the upper portion of the failure envelope) has increased by about this same amount. It was noted from examination of earlier data that most of this change occurred during the first half month of aging at 135°F. Test values obtained for TP-H1141 at 2 in./min and 77°F after 4 mo bulk aging at 135°F are compared with zero time values below.

	After 4 Mo Aging	Zero Aging
Max Stress (psi)	164	144
Max Corrected Stress (psi)	212	196
Strain at Max Corrected Stress (%)	30	41
Strain at Rupture (%)	32	44

3.2 BIAXIAL MECHANICAL PROPERTIES AND AGING

The biaxial tensile data for TP-H1141 propellant aged to 8 mo at 77° and at 135°F (Tables 2 and 3 and Figure 4) are similar to the uniaxial data. They indicate that stress has increased with age while strain at the low rate and high test temperature (the only test point on the lower portion of the failure envelope, 1.0 in./min

^{*}Development of HTPB Propellant for Ballistic Missiles, Quarterly Progress Report No. 6, AFRPL-TR-73-96, October 1973.

at 77°F) is decreasing. The upper portion on the curve is apparently thus far unaffected by the aging of the propellant. Stress relaxation tests of TP-H1141 propellant aged 4 mo at 135°F (Table 4 and Figure 5) show it to have a higher relaxation modulus than at zero time.

Preliminary analysis of aluminum content effects on ballistic efficiency was recently transmitted to the Project Engineer. It is recommended that AFRPL-generated BATES motor data be included in the final analysis in order to provide statistically valid conclusions.

4.0 PHASE III--DEMONSTRATION

Effort for Phase III has been completed.

5.0 PHASE IV--AGING

The TU-775/03 motor had a complete 80°F set of normal and stress gage readings taken on 17 Apr 1974 prior to raising the conditioning box to 135°F. After six days at 135°F, a set of readings was again taken and the box returned to 80°F (23 April). No changes in the state of cure in the fore end soft spots were noted.

Clip gages and thermocouples were installed in the motor according to Drawing 7U46167-02. On 7 May 1974, the motor was pressurized to 15 psig; both while the pressure was going up and also while the pressure was going down negligible hysteresis effects were shown. Thus, it can be concluded that the propellant behaved elastically and that it is perfectly suitable for use as an instrumented structural test vehicle. Gages were again monitored on 28 Jun 1974 and bore diameter measurements taken at three locations. As specified in the Program Plan, raw data were transmitted to the Project Engineer on memorandum DEVP-74-249. Previous data were sent via DEVP-74-199 and DEVP-74-184.

The TU-775/03 motor is now on a monthly monitoring schedule. Reduced data (to 31 May 1974 for ambient pressure readings on all installed gages) are presented on Table 5. The date and nominal conditioning box temperature are shown at the top

of each column, with gage readings listed in psi for normal and shear gages, inches deflection for the clip gages, and °F for thermocouples. The pre-casting pressure/temperature data were used as the calibration base for the Table 5 information.

During the latter part of May 1974, Thiokol was informed that Aerojet was experiencing difficulty with normal gage output on the Flexible Case Program. It was reported that the two channels of any normal gage were giving different readings, in some cases as much as 60 psi. On 5 Jun 1974, Mr. L. Jensen from Thiokol supported a meeting at AFRPL with Aerojet personnel to review present data on both programs, past history, and to consider methods to prevent or correct the situation.

It was suggested at the above meeting that areas of investigation by Aerojet will include: (1) chemical degradation of the semiconductor strain gages, (2) inconsistency in the two DAS used, (3) cross-talk between circuit boards and/or junction boxes, (4) circuitry problems within the gage and/or junction boxes, and (5) influence of humidity on gage performance.

From the data shown in Table 5, it is not evident that Thiokol is experiencing the same problem; however, future data will be closely examined to detect any problems that might occur. To alleviate the problem, it is essential that Thiokol be kept informed as Aerojet proceeds into their present contract. If the problem does develop in the Thiokol motors, the Aerojet correction and adjustment procedures can hopefully be applied to our data.

HTPB chemical structural aging program data on the TP-H1139 propellant from the TU-775/03 are showing exceptional properties after 10 wk of accelerated aging (ref May 1974 report dated 12 Jun 1974, Contract F04611-71-C-0049).

The TU-775/02 motor was instrumented, and precalibration and zero shift data transmitted on memorandum DEVP-74-249.

With Mr. Roe in attendance, the TU-775/02 motor was cast on 2 July from 4,000 and 5,500 lb batches of TP-H1139. No significant problems were encountered, although water was found in one drum of HTPB polymer after it had been placed in two premixes. Premixing was again started with clean bowls and uncontaminated R-45M from the same lot.

Oxidizer plugged the chute on the first mix, and AP spilled from the tilt station. About 12 lb of AP was replaced with 90 micron AP.

Oxidizer was fed too slowly on the first mix. Currently, the screen is set and timed while it is empty, and then loaded. It either stalls or goes slower.

Procedures will be changed to increase the oxidizer feed rate.

The freeze sample on the first mix prior to IPDI addition showed small AP lumps so the batch was mixed an additional 30 min after normal end-of-mix. The end-of-mix freeze sample was also checked for lumps and found to be perfectly satisfactory. The freeze samples taken before and after curative addition on the second mix also showed no lumps.

End-of-mix viscosities were 6.4 kilopoise, with end-of-mix temperatures of 137° and 134°F. Mix data are shown in Figures 6 and 7.

Laboratory analysis was as follows:

	Mix	Mix
	8867003	8868004
Before Curative Addition		
Total Solids (%)	88.55	88.64
Acceptable (%)	88.65 + 0.2	
Aluminum (%)	20,16	20.28
Acceptable (%)	20.14 ± 0.2	
End-of-Mix		
Total Solids (%)	88.00	87.99
Acceptable (%)	88.00 ± 0.2	
Aluminum (%)	20.02	19.99
Acceptable (%)	20.00 ± 0.2	

Loaf samples were cast from each mix and liner boxes were cast from the last mix.

At 2030 hours on 2 July, the motor was put into cure for 216 hours at 135°F.

Test plans for the TU-775/02 and TU-775/03 have been updated to reflect contract changes specified by P00008. Copies were sent to the Project Engineer with memorandum DEVP-74-249.

6.0 PHASE V--SCHEDULE

The documentary movie effort has been completed except for preparation of a caption sheet, and assembly of master and work print for delivery during the next period.

The TU-775/02 will be cured, core and fins will be removed, and X-ray inspected during the next period. Clip gages and thermocouples will be installed in the bore, and the post-loading pressure cycle to 15 psig at 80°F will be conducted. The TU-775/03 will be monitored monthly.

As agreed during Mr. Roe's visit to the Wasatch Division on 1-3 July, a final report outline will be prepared and sent to him for comment.

Delivery to Ogden ALC (upon direction by the PCO) of both motors is set for 1 Nov 1974. It is presumed that GFE Boeing harnesses and a transporter can be made available then.

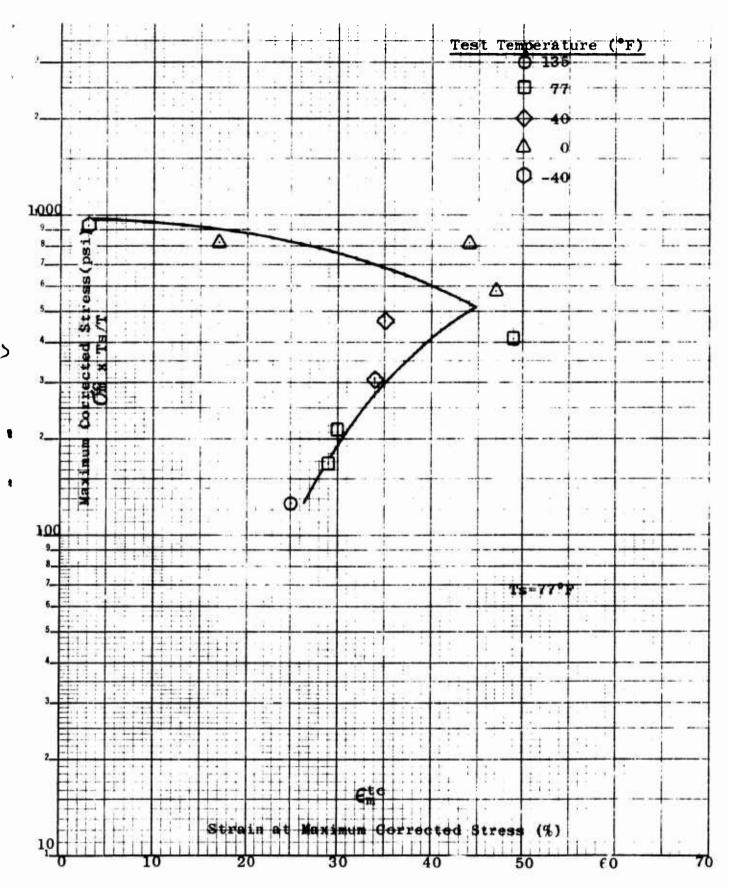


Figure 1. Uniaxial Failure Envelope of TP-H1141 Propellant, Mix 8627001, Aged 4 Months at 135°F

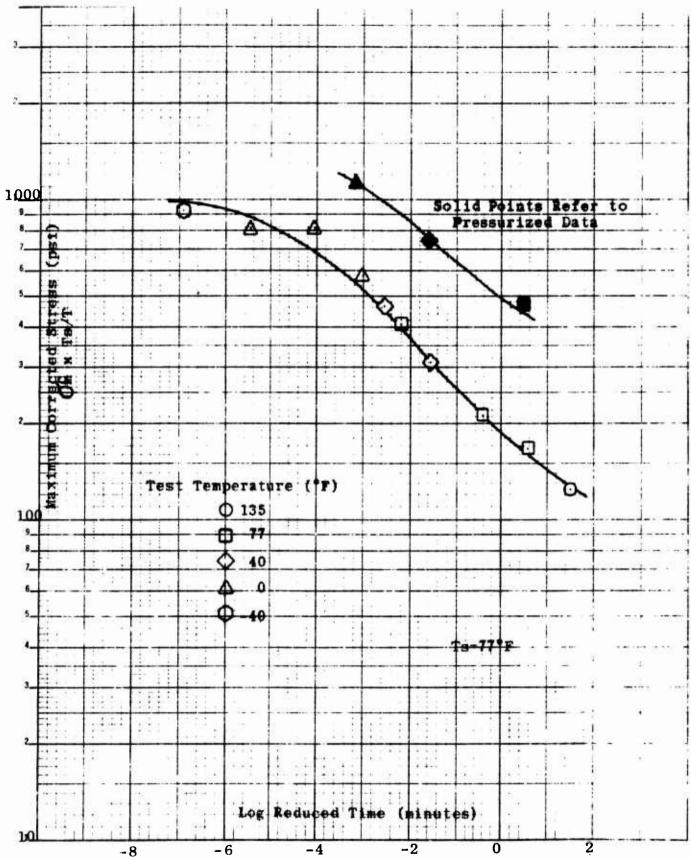


Figure 2. Maximum Stress Master Curve for TP-H1141 Propellant, Mix 8627001, Aged 4 Months at 135°F

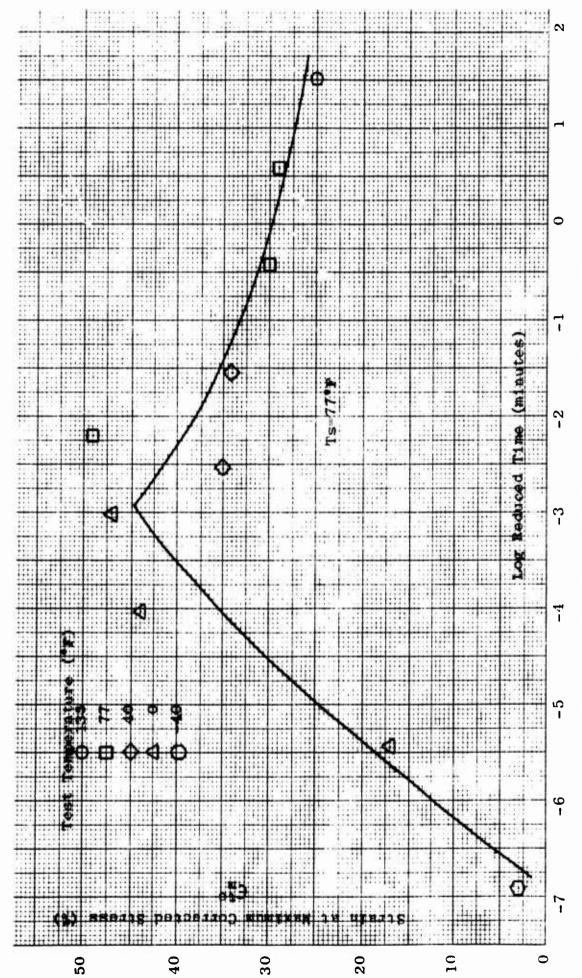
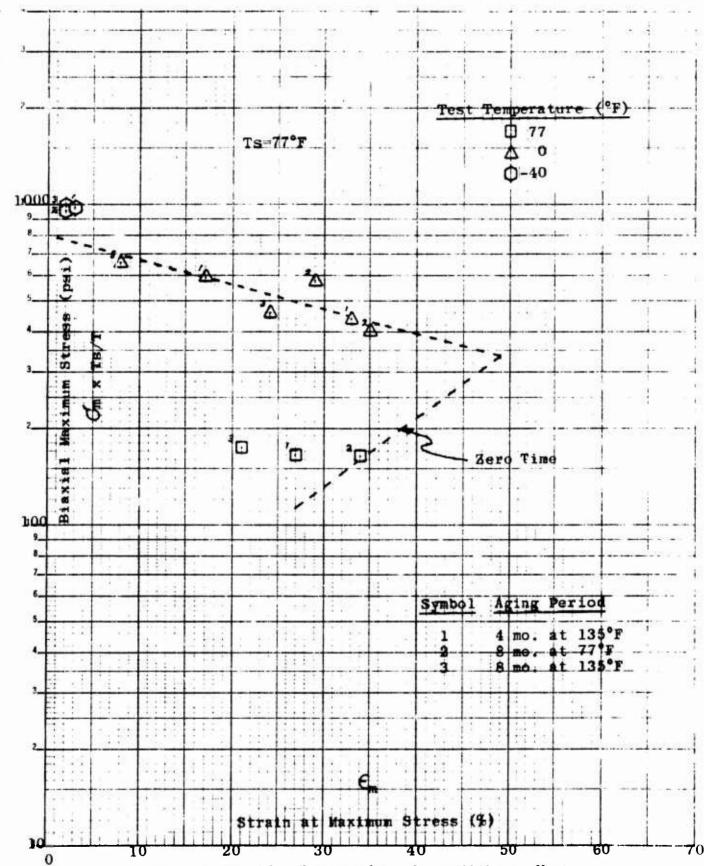
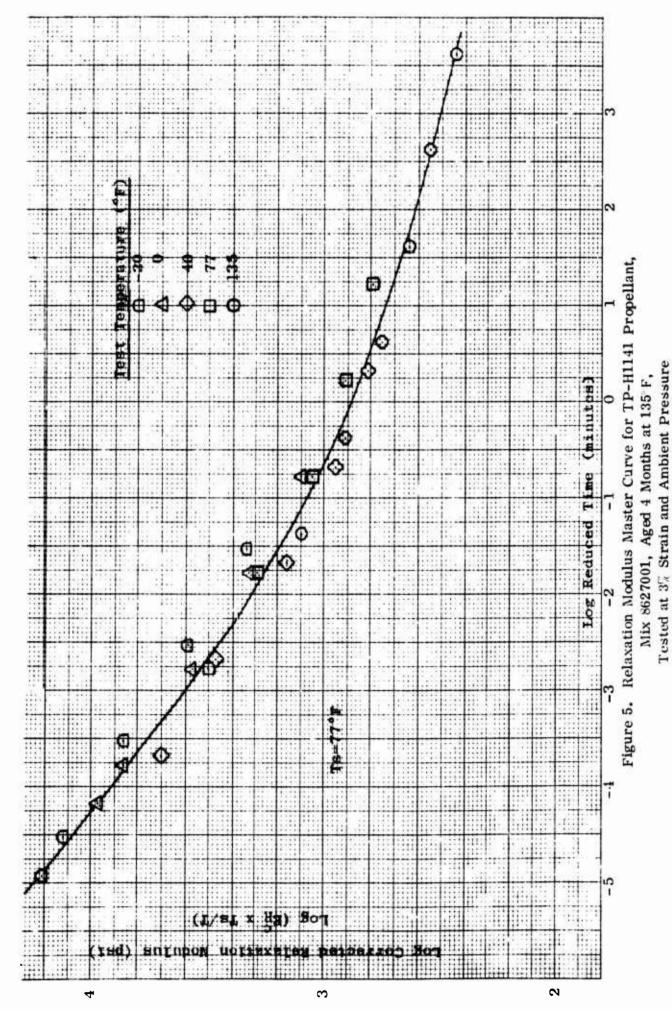


Figure 3. Uniaxial Strain at Maximum Stress Master Curve for TP-H1141 Propellant, Mix 8627001, Aged 4 Months at 135 F



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Figure 4. Biaxial Failure Envelope of TP-H1141 Propellant, Mix 8627001, Aged to 8 Months



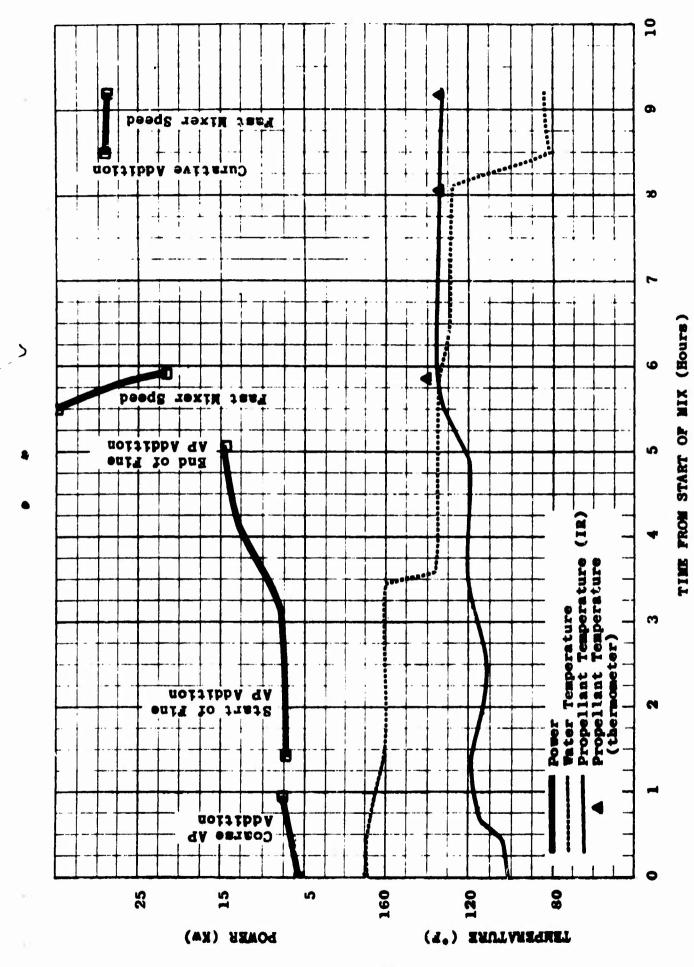
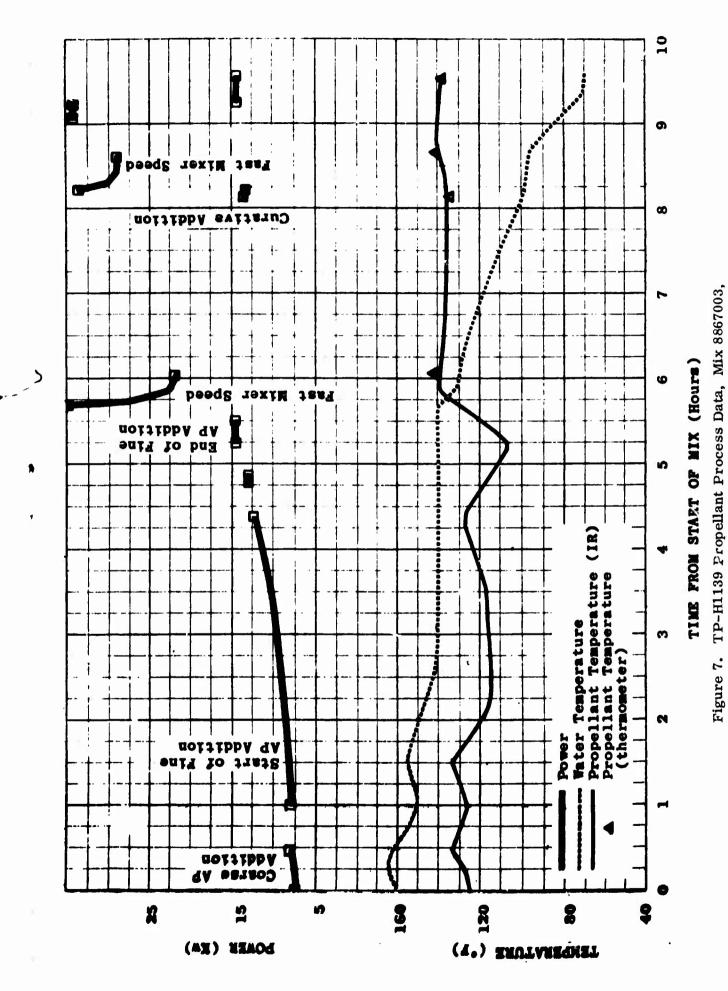


Figure 6. TP-H1139 Propellant Process Data, Mix 8867004, 5,500 Lb, Made 2 July 1974



4,000 Lb, Made 2 July 1974

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TABLE 1

UNIAXIAL TENSILE PROPERTIES OF TP-H1141 PROPELLANT, Mix 8627001, Aged 4 Months at 135°F

Cpsi) 125 167	45
	212 408
311	290 434
579 815 818	496 698 701
927	725
469	469
745	694 7
1,120	

TABLE 2

BIAXIAL TENSILE PROPERTIES OF TP-H1141 PROPELLANT,
Mix 8627001, Aged at 77°F

Aging Time (mo)	Test Temp (°F)	Crosshead Rate (in/min)	りm (psi)	Om Ts/T (psi)	€ _m (%)	Log Time (min)	Log a _T	Log t/aT (min)
0	77 `	0.1 1.0	118 144	118 144	30 33	0.65 -0.31	0	0.65 -0.31
	40	1.0	192	205	40	-0.22	1.15	-1.37
	0	1.0 10	377 496	441 580	9 6	-0.87 -2.05	2.8	-3.67 -4.85
	-40	10	798	1020	2	-2.52	4.7	-7.22
	Pressu	rized at 80	00 psi:					
	40	1.0	487	521	25	-0.43	1.15	-1.58
	0	1.0	669	783	12	-0.74	2.8	-3.54
1	77	1.0	144	144	31	-0.33	0	-0.33
	0	1.0 10	337 505	394 591	40 10	-0.22 -1.82	2.8	-3.02 -4.62
	-40	10	743	1220	7	-1.98	4.7	-6.68
2	77	1.0	158	158	34	-0.29	0	-0.29
	0	1.0 10	327 470	383 550	41 32	-0.21 -1.32	2.8	-3.01 -4.12
	-40	10	717	918	4	-2.22	4.7	-6.92
4	77	. 1.0	165	165	35	-0.28	0	-0.28
	0	1.0 10	366 508	427 593	37 27	-0.26 -1.39		-3.06 -4.19
	40	10	765	978	3	-2.35	4.7	-7.05
8	77	1.0	164	164	34	-0.29	0	-0.29
	0	1.0 10	343 491	400 573	35 29	-0.28 -1.36		-3.08 -4.16
	-40	10	745	953	2	-2.52	4.7	-7.22

TABLE 3

BIAXIAL TENSILE PROPERTIES OF TP-H1141 PROPELLANT,
Mix 8627001, Aged at 135°F

Aging Time (mo)	Test Temp (°F)	Crosshead Rate (in/min)	$C_{\rm m}$ (psi)	Om Ts/T (psi)	€ _m (%)	Log Time (min)	Log a _T	Log t/a _T (min)
0	(See	77°F Aging	Data, T	able 2)				
1/2	77	1.0	164	164	35	-0.28	0	-0.28
	0	1.0 10	362 502	422 586	35 12	-0.28 -1.74	2.8	-3.08 -4.54
	-40	10	726	929	3	-2.35	4.7	-7.05
	77	1.0	155	155	26	-0.41	0	-0.41
	0	1.0 10	329 443	384 517	19 16	-0.55 -1.62	2.8	-3.35 -4.42
	-40	10	714	913	3	-2.35	4.7	-7.05
2	77	1.0	166	166	32	-0.32	0	-0.32
	0	1.0 10	363 498	424 581	35 9	-0.28 -1.87	2.8	-3.08 -4.67
	-40	10	743	950	4	-2.22	4.7	-6.92
4	77	1.0	167	167	27	-0.39	0	-0.39
	0	1.0 10	377 504	440 588	33 17	-0.31 -1.59	2.8	-3.11 -4.39
	-40	10	763	976	3	-2.35	4.7	-7.05
8	77	1.0	174	174	21	-0.50	0	-0.50
	0	1.0 10	394 563	460 657	24 8	-0.44 -1.92	2.8	-3.24 -4.72
	-40	10	775	991	2	-2.52	4.7	-7.22

TABLE 4

RELAXATION MODULUS OF TP-H1141 PROPELLANT,
Mix 8627001, Aged 4 Months at 135°F,
Tested at 3% Strain and Ambient Pressure

Test Temp (°F)	Log Time (min)	ER (psi)	ER x Ts/T (psi)	Log ER x Ts/T (psi)	Log a _T	Log t/a _T (min)
20	0.10	13300	1,6200	4.21	9.75	4 02
-20	-2.18 -1.78	10900	16200 13300	4.21	2.75	-4.93 -4.53
	-0.78	5910	7210			-4.55 -3.53
	0.22	3170		3.86		-3.53 -2.53
			3870	3.59		
	1.22	1780	2170	3.34		-1.53
0	-2.18	8080	9430	3.97	2.00	-4.18
	-1.78	6150	7180	3.86		-3.78
	-0.78	3170	3700	3.57		-2.78
	0.22	1840	2150	3.33		-1.78
	1.22	1090	1270	3.10		-0.78
40	-2.78	4640	4980	3.70	0.90	-3.68
-10	-1.78	2770	2970	3.47		-2.68
	-0.78	1340	1440	3.16		-1.68
	0.22	831	892	2.95		-0.68
	1.22	598	642	2.81		0.32
77	-2.78	3140	3140	3.50	0	-2.78
	-1.78	1930	1930	3.29	U	-1.78
	-0.78	1130	1130	3.05		-0.78
	0.22	808	808	2.91		0.22
	1,22	622	622	2.79		1.22
	1,22	022	022	2.15		1.22
135	-2.78	1380	1250	3.10	-1.40	-1.38
	-1.78	895	808	2.91		-0.38
	-0.78	616	556	2.75		0.62
	0.22	479	432	2.64		1.62
	1.22	394	356	2.55		2.62
	2.22	305	275	2.44		3.62

TABLE 5

>

TU-775/03 GAGE DATA READOUT (Sheet 1 of 3) Normal Gage Readout (pat)

5-31-74 80° F -3.6 •13.3 -5.2 -10.1 -7.8 -5.6 -1.1 -1.8 4.3 0 E 4 -3.2 -0.7 -1.2 5-24-74 80° F -0.5 -0.5 -3.7 6.0 -1.0 -3.4 2.6 6.7 -3.0 -0.9 5-17-74 -3.4 -3.4 -3.5 -13.5 -6.1 -11.4 -8.5 -6.0 -2.0 -2.0 -4.4 -4.4 1.6 11.8 -5.3 5-10-74 80° F -3.9 -3.1 -13.0 -10.0 -7.8 -6.0 -0.6 -1.3 4.5 6.0 -1.1 -1.1 -0.3 -0.4 -1.3 5-2-74 80° F -1.2 -1.4 -3.4 6.3 4.7 -5.9 0.1 -3.9 6.4 8.3 5.2 -3.3 -0.7 0.4 5-1-74 80° F -2.5 -2.1 -12.6 -6.5 -1.0 7.7 -1.1 0.3 -6.4 -3.7 4-30-74 80° F -4.0 -2.7 -6.0 -13.3 -9.0 -8.1 -5.7 -0.4 -1.3 4.1 -3.8 1.2 -0.4 7 1-29-74 70° F -3.5 -2.2 -13.0 -6.0 -7.9 -6.2 -1.5 -5.0 4.5 8.5 -5.3 -1.2 -7.2 -0.4 -3.8 -1.5 135° F -2.6 -1.2 -5.7 90° F -3.4 -2.7 -12.2 -1.5 9.0-1-16-74 80. F -2. 6 -1. 9 -1. 9 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 0 -7. 6 -7. 10 -7. 1 4.4 -5.2 -3.7 -1.5 -1.4 2.7 N10-A N10-B N11-A N11-B N12-A N9-A N9-B | CARP | N1-A | N1-B | N2-A | N2-B | N3-A | N3-B | N3-A | N3-B | N3-B | N5-A | N5-B | N6-B N6-B N7-A N7-B N8-A N8-B

TABLE 5

TU-775/03 GAGE DATA READOUT (Sheet 2 of 3)

1 g	1-17-74 80° F	4-23-74 135° F	4-29-74 70° F	4-30-74 80° F	5-1-74 80° F	5-2-74 80° F	5-10-74 80° F	5-17-74 80° F	5-24-74 80° F	5-31-74 80° F
	9.0	1.3	9.0	0.7	7.0	0.7	9.0	9.0	9.0	9.0
	0.4	4.0	7.0	9.0	0.5	0.4	0.4	0.4	0.4	0.4
	1.0	1.8	8.0	0.9	1.4	1.1	1.0	6.0	0.9	0.9
	2.6	3.2	3.2	3.1	2.8	2.8	3.0	3.0	2.8	3.0
'	-0.8	4.0-	8.0-	-0.8	-0.7	-0.7	8.0-	-0.8	8 -0-	-0.8
	0.4	1.5	0.7	0.7	9.0	0.7	0.7	9.0	9.0	9.0
	0	1.2	0.1	0.1	0.3	0.3	0.2	0.1	0.2	0.1
	0.2	1.0	0	0.1	0.3	0.3	0.1	0.1	0.2	0.1
•	-1.2	-0.7	-1.5	-1.4	-1.2	-1.1	-1.2	-1.2	-1.1	-1.1
•	-1.8	-0.7	-2.3	-2.3	-2.1	-1.9	-2.0	-2.0	-1.9	-1.9
•	-2.5	0.4	-3.6	-3.5	-3.1	-2.8	-3.0	-3.0	-2.7	-2.9
	1.0	-1.0	-0.7	-0.7	-0.6	-0.6	-0.5	9.0-	-0.5	-0.5
•	-4.2	-3.9	-5.9	8.5.	-5.3	-5.0	-5.0	-5.0	4.9	-5.0
•	-3.5	1.0	7.4-7	•	-3.6	-3.4	-3.7	-3.8	-3.3	-3.7
•	-2.3	1.7	4.1	-3.9	-3.2	-2.7	-2.9	-2.9	-2.4	-2.6
	2.2	1.0	2.2	•	2.2	2.1	2.2	2.1	2.1	2.0
	0.4	0.1	0.3	0.3	0.3	0.3	0.4	0.4	0.4	0.5
						Clip Gag	Clip Gage Deflection (in.	.,	ı	
					-0.710	-0.721	-0.712		-0.707	-0.706
					-0.342	-0.339	-0.335	-0.326	-0.326	-0.323
					-0.147	-0.170	-0.118	-0.116	-0.113	-0.139

·Bad reading.

TABLE 5

TU-775/03 GAGE DATA READOUT (Sheet 3 of 3) Thermocouple Readout (F)

5-31-74	28	71	7.8	7.7	79	7.8	g ;	80	7.7	78	80	80	80	79	80	7.8	99	7.6	92	80	80	80
5-24-74 80° F	8	81	81	81	81	81	82	82	81	81	80	79	79	80	82	82	82	80	80	9	80	79
5-17-74 80° F	12	77	7.2	7.7	7.7	7.7	7.7	78	7.2	77	79	7.9	42	78	7.7	7.2	77	3.6	76	79	42	79
5-10-74 80° F	12	78	82	77	78	78	78	80	11	77	80	80	80	79	79	7.7	78	76	76	80	80	80
5-2-74 80° F	8	83	83	82	84	83	83	48	83	83	18	81	80	83	34	83	83	82	81	82	81	:
5-1-74 80° F	79	43	90	79	80	79	80	80	79	90	ŀ	;	1	;	81	42	80	78	78	1	ł	ŀ
4-30-74 80° F	22	73	74	73	73	73	73	73	73	7.4	1	1	1	ł	74	74	73	73	73	1	:	ı
4-29-74 70° F	112	70	11	70	10	11	70	11	72	72	1	1	ł	ł	72	72	72	72	72	1	;	;
4-23-74 135° F	132	133	133	131	133	133	133	134	132	132	ł	;	1	1	134	132	132	129	128	1 }	;	;
4-17-74 80° F	79	79	42	78	42	49	49	80	42	79	1	•	1	;	80	42	7.9	78	78	;	:	:
4-16-74 80° F	78	78	78	78	78	78	78	80	78	78	;	;	1	:	78	7.8	78	78	78	1	:	i
Gage	Ę	T2	ET.	T4	T5	Te	17	T8	T10	T11	T12	T13	T14	T15	T16	T17	T18	T19	T20	T21	T22	T23